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AN EXPERIMENTAL STUDY IN THE MECHANICAL RESPONSE OF POLYMER MODIFIED GEOPOLYMERS

Benjamin Valera

Department of Mechanical Engineering
Rochester Institute of Technology
76 Lomb Memorial Drive
Rochester, NY 14623-5604

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ANGELICA CUBILLOS, 1st Lt, USAF
Work Unit Manager

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Digitally signed by MELLERSKI.ROBERT.C.1021956941
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R. CRAIG MELLERSKI, DR-III
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76 Lomb Memorial Drive
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14. ABSTRACT

The objective of this project was to evaluate the effect of low molecular weight of Poly Ethylene Glycol (PEG), Carboxy Methyl Cellulose (CMC) on metakaolin and F type fly ash sodium based geopolymers in heir mechanical response. It was found that adding 22.5% per weight of a Carboxyl Methyl Cellulose solution to metakaolin geopolymers over a mesh of cheesecloth modifies the elastic modulus producing a plate with enhanced flexibility. This effect was not observed when randomly dispersed fibers were used in the same formulation. The addition of randomly dispersed fibers such as cotton and Poly Vinyl Alcohol increased the bending and compressive strength of geopolymers.

In the case of fly ash geopolymers, no flexibility was observed in the plates and changes in the viscoelastic properties were not detected. The addition of Carboxy Methyl Cellulose and Poly Ethylene Glycol in fly ash geopolymers enhanced the bending strength but the compressive strength decreased.

15. SUBJECT TERMS

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1. SUMMARY

This project pertains to the category entitled Aircraft Operating Surfaces. The goal of the project is to investigate the feasibility of using polymer-modified geopolymers in military applications, such as surfacing airfield facilities. The addition of hydrophilic polymers offers the potential for modifying the setting and flexural properties of geopolymers, consequently opening an array of new applications where geopolymer properties such as strength and fire resistance are combined with resilience and ductility.

The specific objectives are: 1) Evaluate the effect of low molecular weight of polyethylene glycol (PEG), carboxymethyl cellulose (CMC) on metakaolin and Class F fly ash [1] geopolymers using a sodium solution as activator. 2) Evaluate the effect of cellulose (cotton) and polyvinyl alcohol (PVA) based fibers in the viscoelastic response, flexural and compressive strength of polymer modified geopolymers. 3) Establish an ongoing research relationship with the Air Force Research Laboratory, Airbase Technology Division (AFRL) for collaboration in developing geopolymers.

It was found that adding 22.5% per weight of a CMC solution to metakaolin geopolymers over a mesh of cheesecloth modifies the elastic modulus producing a plate with enhanced flexibility. This effect was not observed when randomly dispersed fibers were used in the same formulation. The addition of randomly dispersed fibers such as cotton and PVA increased the bending and compressive strength of geopolymers.

In the case of fly ash geopolymers, no flexibility was observed in the plates and changes in the viscoelastic properties were not detected. The addition of CMC and PEG in fly ash geopolymers enhanced the bending strength but the compressive strength decreased.

2. INTRODUCTION

Geopolymers constitute a new class of technological materials with great potential for civil and military infrastructure. Geopolymers exhibit some unique engineering properties, such as high early compressive strength, low thermal conductivity, high thermal stability, non-flammability, and strong bonding to metallic and concrete substrates. The mix of properties make geopolymers as ideal candidates for Air Force applications, including novel materials for runways, fire resistant coatings, liners, geotextiles and high performance materials in facilities

These inorganic polymeric materials are produced by a polycondensation reaction between an aluminosilicate and an activating solution at temperatures below 100 °C, including room temperature. In this reaction, tetrahedrons of SiO_2 and AlO_4 are linked together by oxygen bridges, forming a complex three dimensional network which requires Na^+, K^+ or Ca^+ ions to balance their charge. The product is a monolithic material whose properties are comparable or better than materials based on ordinary Portland cement (OPC).

Several factors determine the final properties of the geopolymer. These include the type of alkali, its concentration, the type of aluminosilicate and the curing process. A number of international scientific groups have been experimenting with the various influences of these factors on the final properties. The most studied properties include the mechanical, thermal, resistance to sulfate attack, fire resistance and encapsulation of radioactive waste.

Geopolymers are inorganic polymeric materials, also known as polysialates, which consist primarily of silica tetrahedrons bonded to alumina tetrahedrons with sodium or potassium ions balancing the charge of the structure. They belong to a new class of amorphous aluminosilicates that can be cured at low temperatures, including ambient temperature, and atmospheric pressure. These materials were first recognized by V.D. Glukhovsky as “soil cements”. He claimed that in ancient concretes, substances derived from the reaction of aluminosilicates in high alkaline conditions exhibited superior properties due to the coexistence of CSH phase and alkali aluminosilicate hydrate phases [2].

French chemist Joseph Davidovits is credited with the discovery of geopolymers in the 1970’s. His motivation for this research was to develop non-flammable, non-combustible polymeric materials. As a result of this research, J. Davidovits registered a series of patents for the synthesis and application of geopolymers with commercial purposes. For this reason during the 1970’s and 1980’s development in geopolymers was unknown for most of the scientific community. In 1991 Davidovits disclosed this technology and summarized the work done until then, this attracted immediate attention of scientist and engineers in Europe and Australia [3].

Since then several research groups all over the world have focused their efforts in various aspects of geopolymer science and technology. The group at the University of Melbourne, led by J. van Deventer and J. Provis, and at the University of Illinois led by W. Kriven have worked in gaining fundamental knowledge on the chemistry and physics of the geopolymerization reaction [4,5]. The group led by A. Palomo and M.T. Fernandez [6] in Spain has made significant contributions in the use of fly ash as raw material for the synthesis of geopolymers and their use in civil engineering applications. Another group in Australia led by B.V. Rangan investigated the long term stability of geopolymers and their flexural strength response while reinforced with steel

bars [7]. In the United States, another group led by P. Balaguru at Rutgers University investigated the use of carbon fibers in geopolymers for aircraft applications [8].

As mentioned before, two of the most relevant properties of geopolymers are their high early compressive strength and their stability at high temperatures. A geopolymer can reach between 20 and 36 MPa in 24 hours and up to 83 MPa in 28 days as compared to OPC which reaches approximately 48 MPa in the same time frame. OPC starts deteriorating at 450 °C while geopolymers remain stable up to 1000 °C.

Although this technological research has mostly been conducted in Europe and Australia, it is an emerging field of experimentation in the United States. Several areas offer opportunities for research and development, particularly in the usage of this technology as it applies to the military. One of the primary uses of this technology would be in the surfacing of airfield facilities.

Runways are constantly subject to impact loads and stresses due to normal operation of aircrafts. This causes permanent deformation on the surface of the runway and safety issues to the air facility. The blast of the jet engines is another cause of deterioration of the surfaces. Most construction materials do not have the capacity to resist the thermal impact, and eventually they spall. Water and deicers are two other factors that cause the surface of runways to deteriorate. This project will work toward developing new materials that can overcome these problems. Geopolymers are good candidates for this application.

Metakaolin has been one of the preferred sources for geopolymer synthesis due to its low content of impurities and high reactivity. Geopolymers based on Metakaolin have been fully characterized and their properties found to be predictable [9]. Other research groups have explored several types of industrial by-products rich in reactive silica and alumina, such as fly ash, slag and mining tails, with the goal of developing a cost effective material. Geopolymers synthesized from industrial byproducts require more care due to the presence of impurities and other oxides, in particular the amount of CaO.

As pointed out by Teixeira-Pinto [10], published research does not mention the difficulties associated with the processing of geopolymers. While small scale processing is controllable, larger scale manufacturing may result in variables not seen at the laboratory level. The change of the rheology is one of the observed problems in large scale preparation which requires detailed attention. The suggested method to prepare geopolymers is to slowly add the aluminosilicate to the activating solution using a shear mixer. However, as the reaction proceeds the viscosity changes and hinders further mixing. For this reason research into rheology modifiers is desirable. Although this project is not oriented towards rheology modifiers, this experimental work can provide an insight to the solution for this problem. This could lead to another project in collaboration with the Air Force Office of Scientific Research.

Previously, the project investigator (PI) conducted a study to evaluate the effect of polymers containing basic O^- or ether linkages which under strong alkaline conditions will react with the uncured geopolymer [11]. The goal of the study was to evaluate their effect upon the rheological properties of geopolymers. The addition of low molecular weight polyethylene glycol (PEG) and

carboxymethyl cellulose(CMC) to the geopolymer paste not only modified the viscosity, but also added elasticity to the cured geopolymer. The resulting paste was pliable and remained in this state long enough for the paste to be easily spread into the molds. After curing, some of the plates showed remarkable flexibility as seen in Figure 1.

Figure 1. Typical Appearance of a Metakaolin Geopolymer Modified with CMC

3. ASSUMPTIONS AND PROCEDURES

3.1. Raw Materials

Commercial grade metakaolin (Metamax, BASF) and Class F fly ash (Bowen, Boral Materials Technology) were used as primary sources of reactive alumina. Both materials were used as received without any further processing.

Table 1 presents this chemical composition in terms of main oxides as provided by the suppliers

Table 1. Chemical Composition of Aluminosilicates in Mass Percentage

Oxide	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO
Metakaolin	53.0	43.8	0.43	0.12	0.02
Fly Ash	55.4	27.6	7.2	2.7	1.2

Aside from their chemical compositions, both materials were characterized for their particle size and distribution in a Beckman Coulter LS particle size analyzer. Figure 2 and Figure 3 present these distributions.

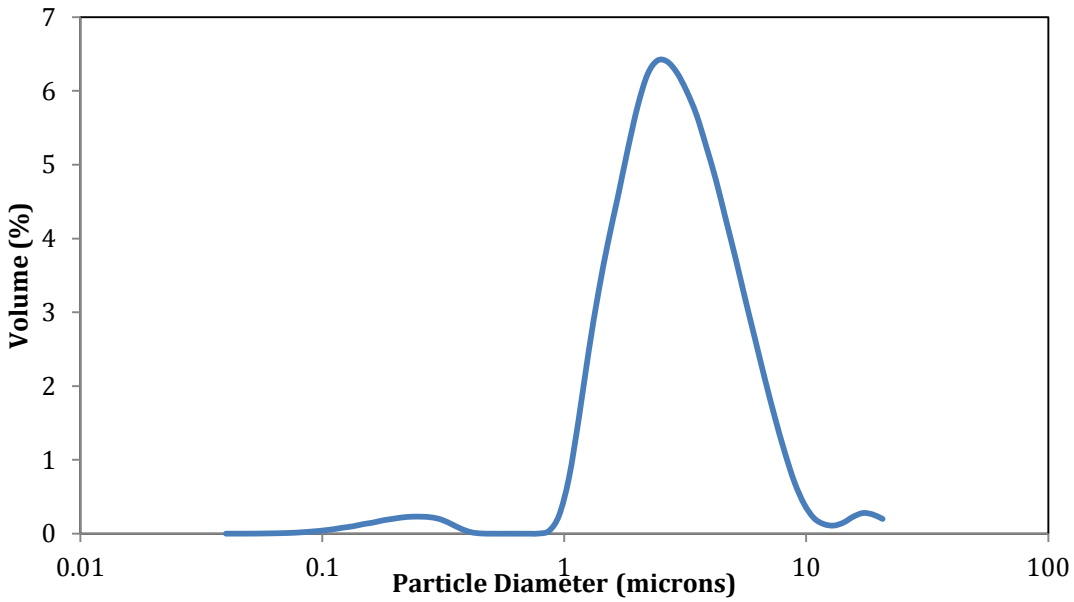

Figure 2. Particle Size Distribution for Metakaolin

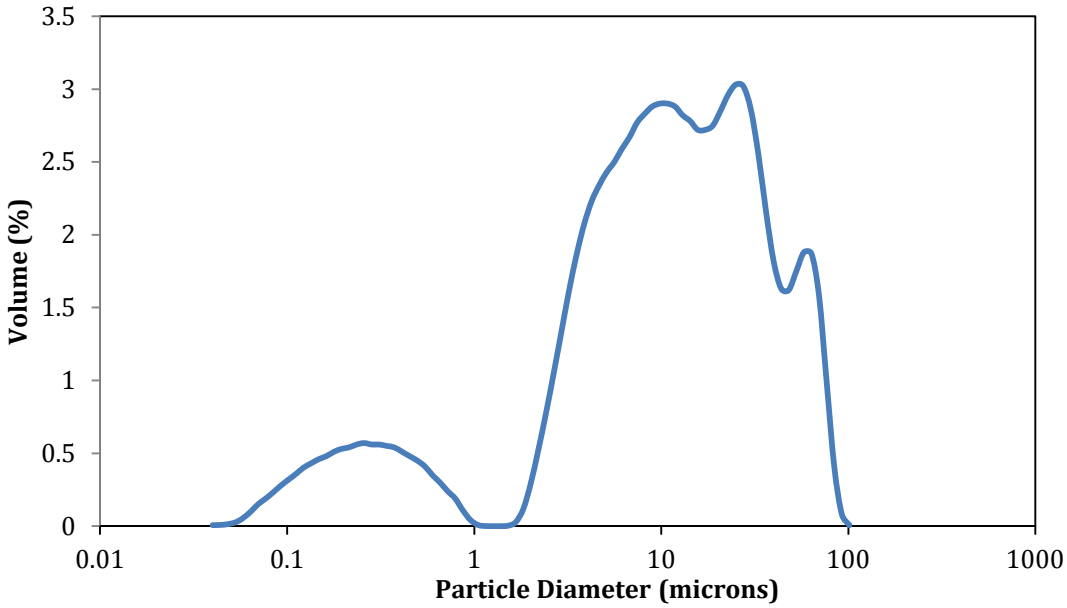

Figure 3. Particle Size Distribution for Fly Ash

This study showed that the metakaolin particles are finer and less dispersed than the fly ash particles. Metakaolin particles have a median size of 3.5 μm with a range between 1 and 10 μm. Fly ash particles have a median size of 14 μm with a range between 1 and 100 μm.

The main source of soluble silicate was a D Type Sodium Silicate (PQ Corporation) with a SiO_2/Na_2O weigh ratio of 2. In some formulation amorphous silica in powder form (Aerosil 300, Evonika Degussa Corporation) was used to modify the SiO_2/Na_2O .The source of soluble alkali was a 40 wt.% technical grade NaOH solution (Brainerd Chemical Company). This solution was diluted in distilled water to achieve a 15 molal solution.

The soluble polymers were polyethylene glycol (Pfaltz & Bauer), MW. 200 and carboxymethyl cellulose, (Across Organics) MW. 90000 .

3.2. Procedures

Metakaolin base geopolymers were prepared by mixing 110 grams of 15 molal NaOH solution with 220 grams of D type sodium silicate. Both liquids were mixed for 20 minutes and then left to cool to room temperature. The molar ratios of this activating solution were $SiO_2/Na_2O = 1$ and $H_2O/Na_2O = 10.7$.

330 grams of metakaolin were slowly added to the activating solution in batches of approximately 50 grams. Addition of each batch was followed by 2 minutes of mixing at low speed in a Hobart mixer. The mixing continued after the addition of the last batch until homogeneous slurry was obtained.

The slurry was casted in acrylic molds of 1 × 1 × 6 inch, vibrated for 10 minutes and covered with plastic to prevent moisture loss. The covered molds were left at room temperature for 1 hour and then cured at 65 °C for 24 hours.

The polymer solutions were prepared prior to the mixing of the aluminosilicate and the activating solution. The first polymer tested, CMC was found to be insoluble in the activating solution and, therefore, it had to be dissolved in distilled water first. The CMC solution was prepared by warming the distilled water to 65 °C and slowly adding the polymer while being stirred. The solutions prepared had 0.5%, 1.0% and 1.5% weight percent of CMC in water. Once the CMC was dissolved in warm water it was allowed to cool to room temperature and then added to the activating solution. The total amount of polymer solution added was 22.5% per weight of the activating solution. This amount was chosen based on the aforementioned study conducted by the PI. Both solutions were mixed prior to use.

PEG was found to be soluble in the activating solution. For this case PEG was added to the activating solution so its total amount represented 0.5%, 1.0% and 1.5% percent per weight of the total solution. However, in this formulation 281 grams of metakaolin was added to the solution since it yielded the best workability.

Class F Fly ash reacts different than metakaolin, for this reason these formulations had to be adjusted. After several trial and error experiments it was found that good workability could be obtained by mixing 50 grams of the 40% NaOH solution, 100 grams of D type sodium silicate and 33.8 grams of distilled water. This solution had $SiO_2/Na_2O = 1$ and $H_2O/Na_2O = 14.1$. This solution was prepared following the same procedure as for metakaolin geopolymers. Once the solution was prepared 300 gr of fly ash were added slowly and mixed until homogeneous slurry was obtained.

When the organic polymers were added to the Fly Ash formulations, CMC and PEG were dissolved as previously described with one difference. Both polymers were dissolved in the distilled water used for the activating solution.

Plates of approximately 10 cm wide × 15 cm long × 1 mm thick were prepared for Dynamic Mechanical Analysis (DMA). A small amount of slurry was uniformly spread over a layer of cheesecloth using a plastic roller. A second layer of cheesecloth was put on top of the slurry and covered with a plastic film. The plate was hand press to remove air pockets and then left to mature for 1 hour at room temperature. After this the plate was pressed for 4 hours at 65 °C and 1 metric Ton of pressure.

For compressive and bending strength measurements the samples were left at room temperature for 7 days. A minimum of 3 samples were tested and their average is reported here.

3.3. Equipment

All the geopolymer mixes were prepared in a Hobart planetary mixer at the lowest speed. The plates were pressed in a Carver press (model 3851) with heated upper and lower plates.

A MTS model 451G universal testing machine was used for compressive and bending tests. For compressive testing the 1 × 1 × 6 inch beams of cured geopolymer were cut into samples of 1 × 1 × 2 inch using a diamond blade saw. For bending test the beams were placed in a three point bending fixture leaving a 4 inch span between the supports. In both experiments the speed of the crosshead was kept constant at 0.02 in/min.

A Perking Elmer DMA 800 was used for DMA. Samples of approximately 0.5 cm wide × 1 cm long × 1.5 mm thick were cut from the pressed plate using a diamond blade and ran in tension mode. The frequencies used were 0.5 and 5 Hz and the temperature range was from -40 °C to 200 °C. All samples were tested in tensile mode.

A JEOL 6400 Scanning Electron Microscope was used to characterize the microstructure of the geopolymers.

4. RESULTS AND DISCUSSION

4.1. Metakaolin Geopolymers

Figure 4 presents the average compressive and bending strengths for metakaolin geopolymers modified with CMC. At 0% CMC loading the average compressive strength was 25.5 MPa and the average bending strength was 3.3 MPa.

With a 0.5% CMC loading both strength decreased significantly only to attain similar strengths to the original formulation at 1.0% CMC loading (26.7 MPa compressive and 3.7 MPa bending). A 1.5% CMC loading didn't affect much the compressive strength but it decreased significantly the bending strength of the samples. In average, the best combination of properties were obtained at 1.0% CMC.

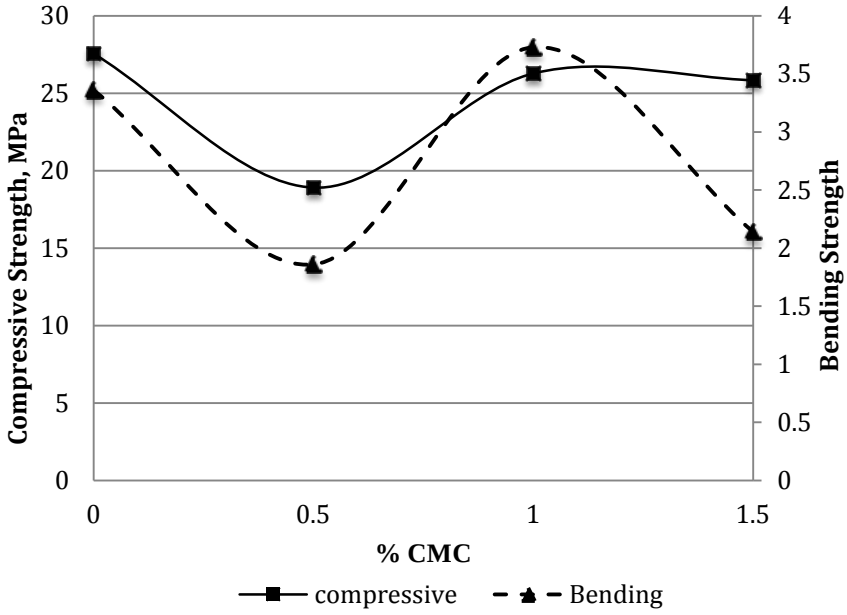

Figure 4. Average Compressive and Bending Strengths for CMC Metakaolin Geopolymer

Figure 5, Figure 6 and Figure 7 present the Elastic Modulus (E'), Viscous Modulus (E") and Tan δ respectively measured by DMA in the range from room temperature to 200 °C. The data presented here corresponds to a frequency of 0.5 Hz since there was no significant difference with the 5 Hz.

Both E' and E" show a smooth decrease with temperature, resembling the behavior of a semi crystalline polymer below the glass transition temperature.

At low temperatures both E' and E" decrease as the amount of polymer is increased to 1.0% only to attain similar values to the base sample once it reaches 1.5%.

Results indicate that adding 0.5% CMC produces a major change in E' and E" at room temperature. These values decreased approximately in the same proportion (about 75%) from 0% to 0.5% CMC. As we increase the loading from 0.5% to 1.0% E' decreases 22% while E" only decreases 12%. What is interesting is that by changing the loading from 1.0% to 1.5% the value of E' increases by a factor of 2.7 while E" does it by a factor of 4.

This behavior can be observed in Figure 7. As the loading of CMC changes from 0% to 1.5% Tan δ increases approximately 75% indicating that E" becomes dominant. For all formulations, at approximately 100 °C, Tan δ increases with temperature, however a transition was not observed in this temperature range

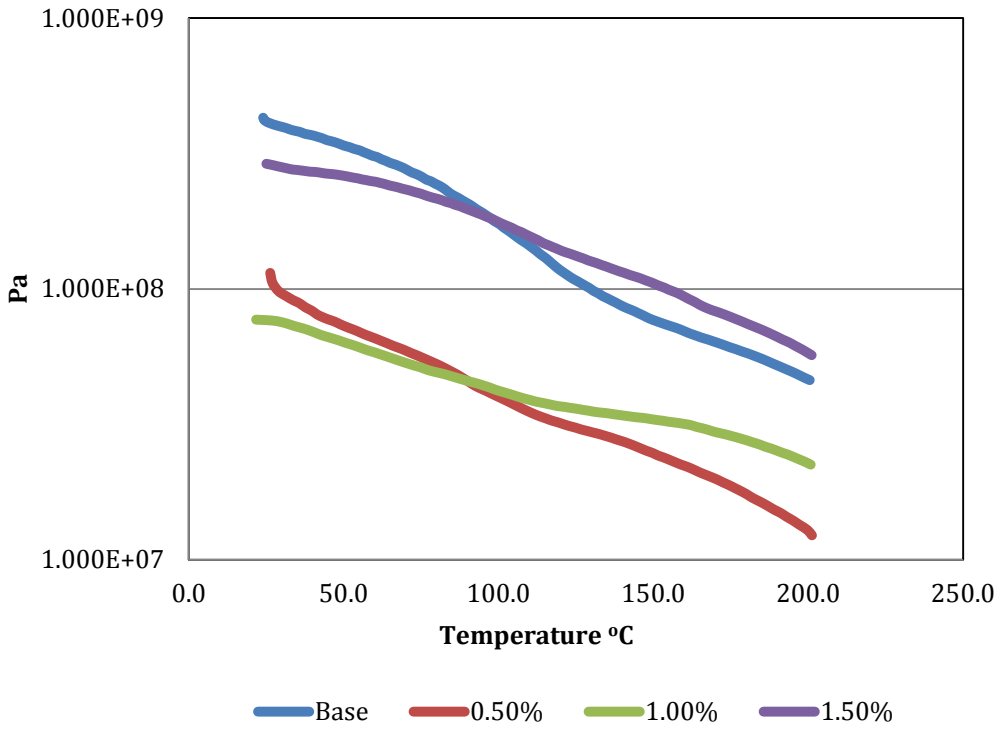

Figure 5. Elastic Modulus (E') for Metakaolin Geopolymers with CMC

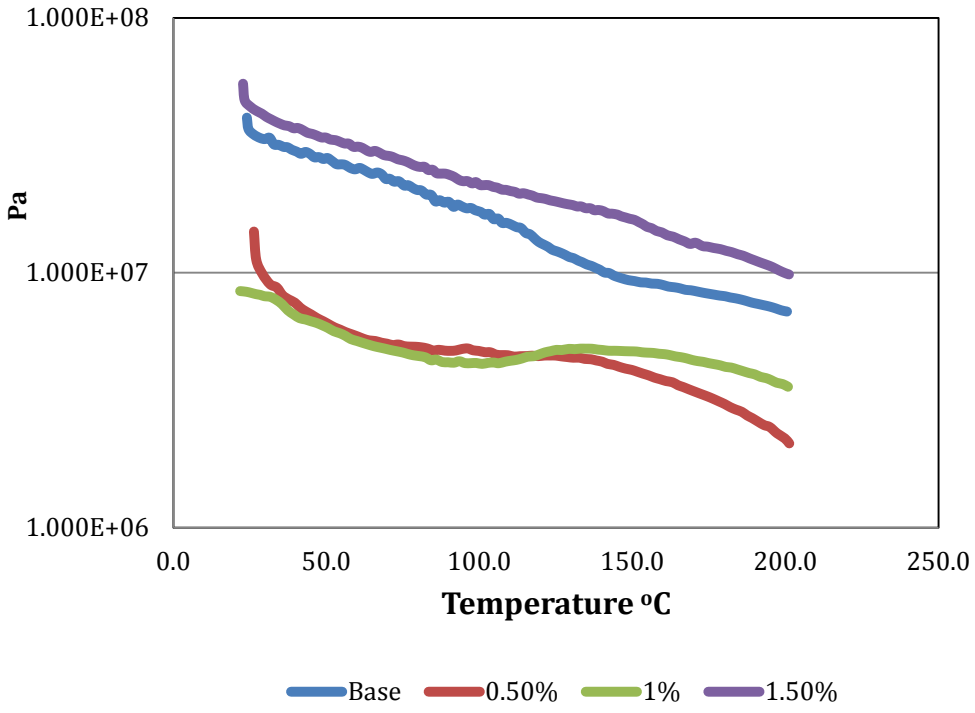

Figure 6. Viscous Modulus (E") for Metakaolin Geopolymers with CMC

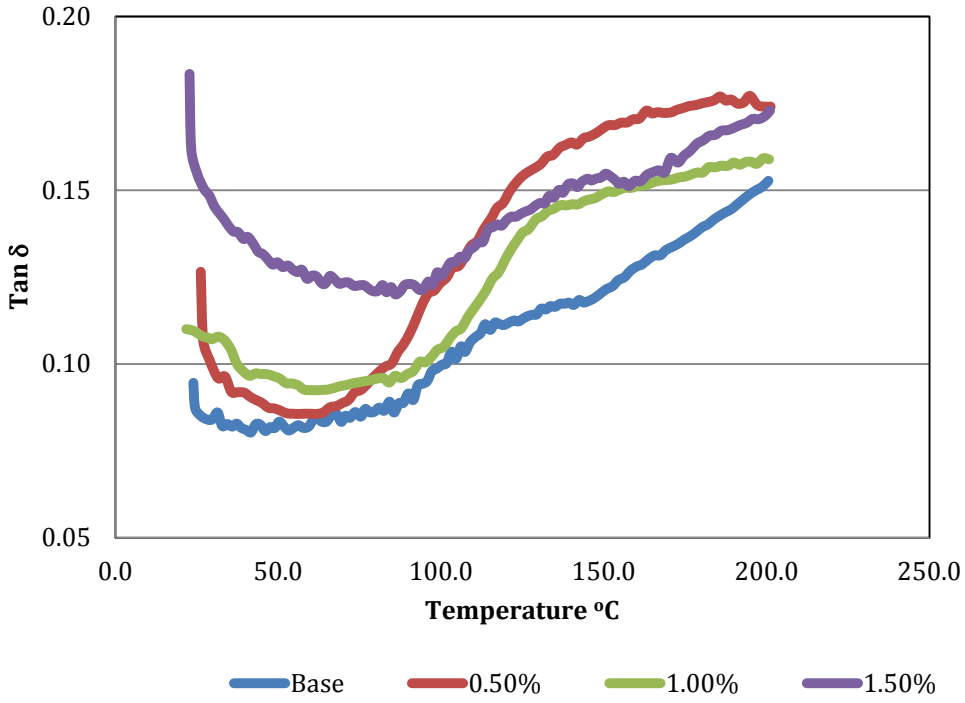

Figure 7. Tan δ for Metakaolin Base Geopolymers with CMC

Figure 8 presents the compressive and bending strength of metakaolin formulations modified with PEG. Adding PEG to metakaolin geopolymers had a significant effect in the compressive strength. The compressive strength increased by a factor of 2 by adding 1.0% PEG while the bending strength only showed a slight increase. In average the best bending strength was obtained by adding 0.5% PEG.

Figure 9, Figure 10 and Figure 11 present the Elastic Modulus (E'), Viscous Modulus (E") and Tan δ respectively for plates with 0% and 1.0% PEG. Samples with 0.5% PEG were too brittle and failed under DMA testing.

Adding 1.0% of PEG caused an increase of E' by a factor of 7 and E" to increase by a factor of 3.4 at room temperature. Physically these plates were very rigid and showed no flexibility at all.

In the case of the base formulation, both E' and E" remained fairly constant up to 120 °C after which they slowly decrease. However for the 1.0% PEG E' decreased at a much faster rate than E" which caused a rapid increase in Tan δ. This suggests that the damping of the geopolymer is increasing.

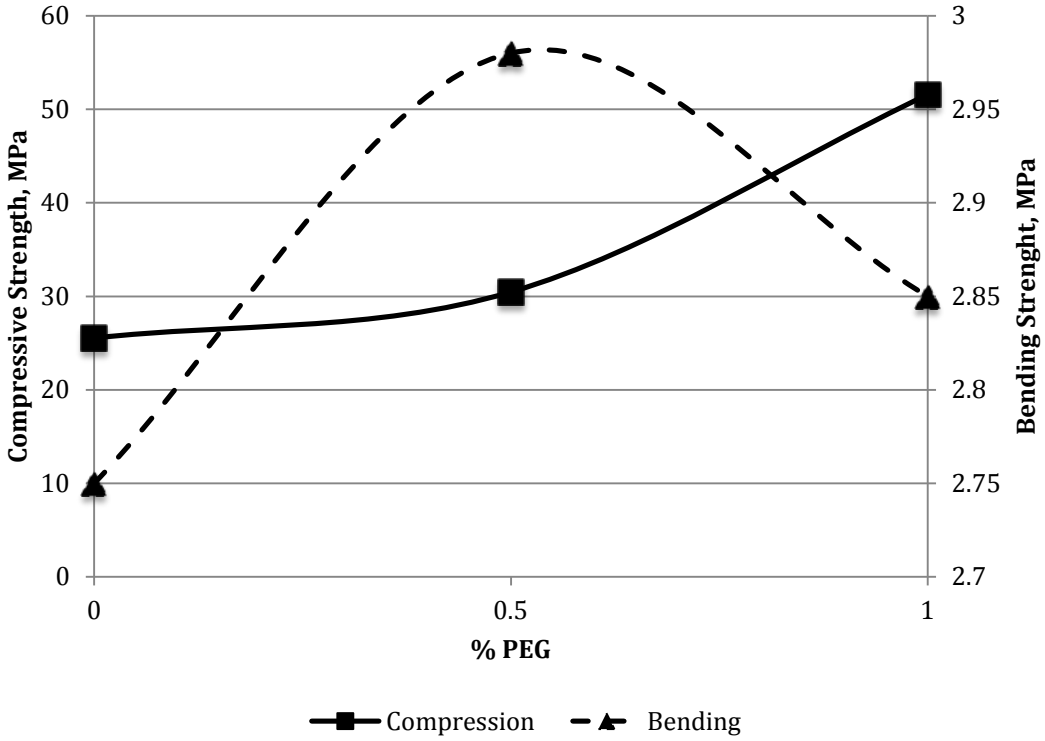

Figure 8. Average Compressive and Bending strengths for PEG Metakaolin Geopolymer

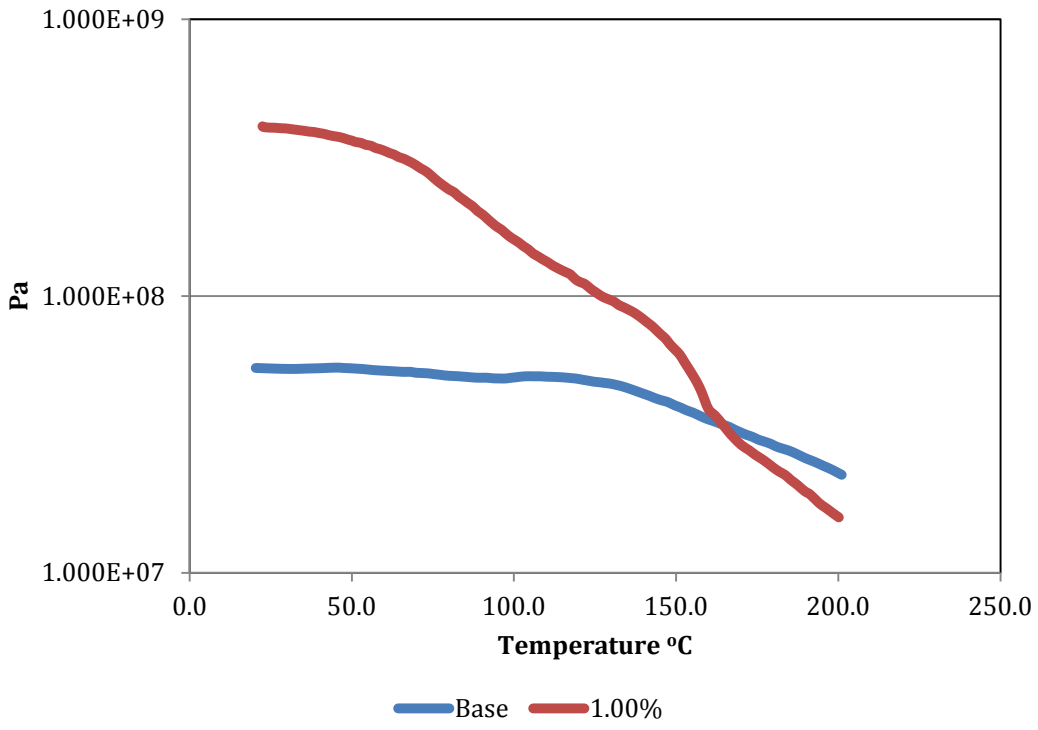

Figure 9. Elastic Modulus (E') for Metakaolin Geopolymer with PEG

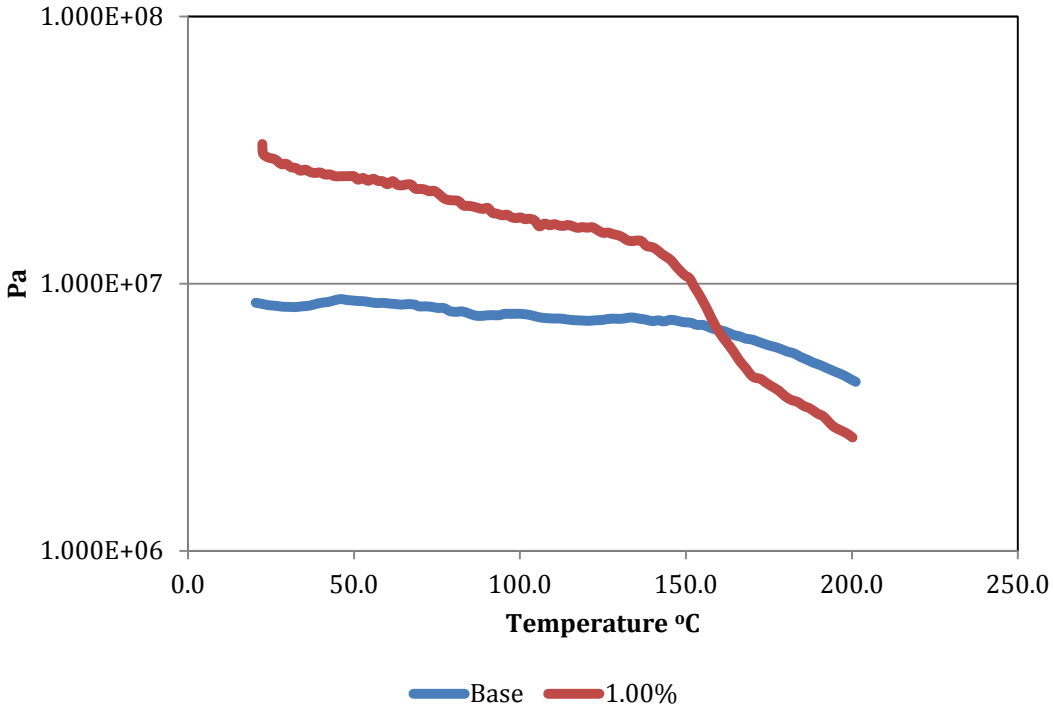

Figure 10. Viscous Modulus (E") for Metakaolin Geopolymers with PEG

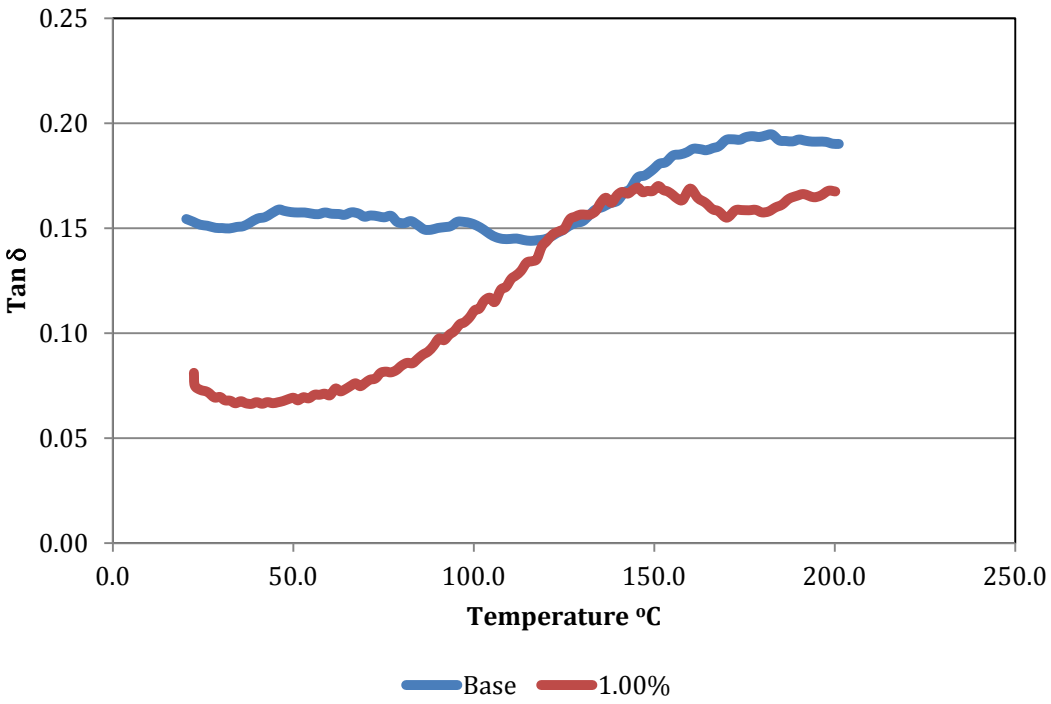

Figure 11. Tan δ for Metakaolin Geopolymer with PEG

Scanning electron micrographs revealed that metakaolin geopolymers are composed of an amorphous phase with unreacted particles of kaolinite (Figure 12). A similar structure was observed in samples with CMC however these samples revealed a needle-like formation between the pores of the amorphous phase (Figure 13). When PEG was added to metakaolin geopolymers, SEM revealed a similar structure to the base formulation (Figure 14).

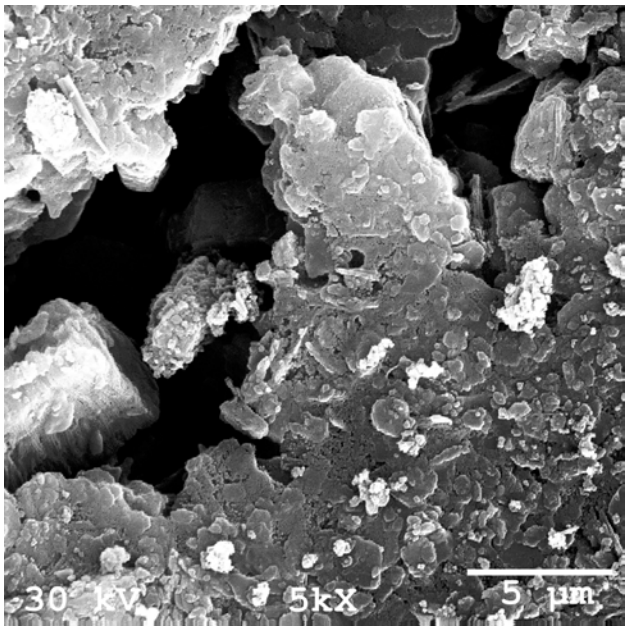

Figure 12. SEM Micrograph of Metakaolin Geopolymer

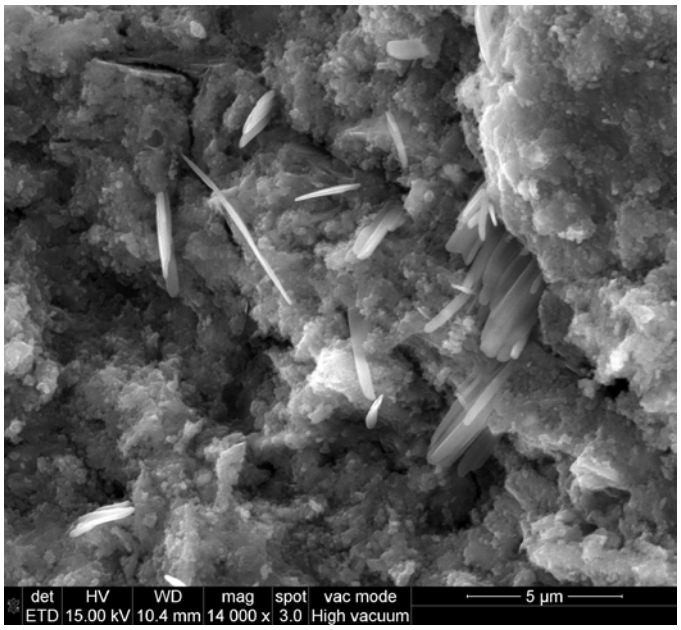

Figure 13. SEM Micrograph of Metakaolin Geopolymer with CMC

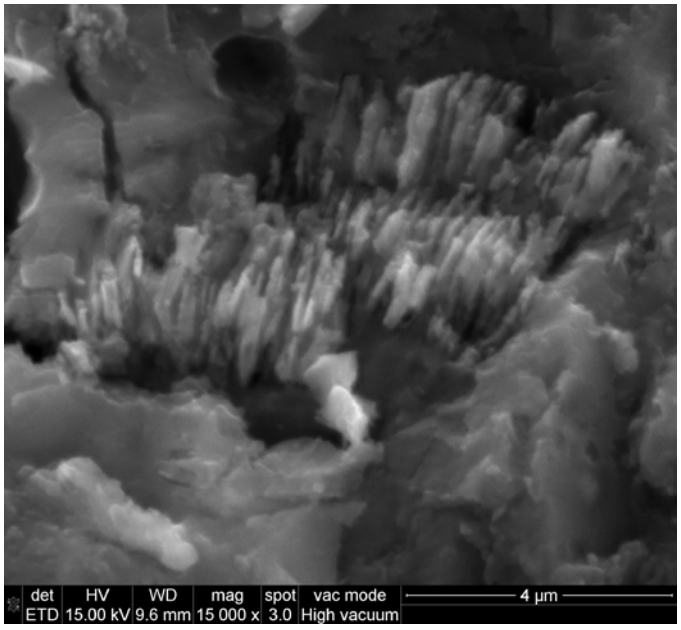

Figure 14. SEM Micrograph of Metakaolin Geopolymer with PEG

4.2. Fly Ash Geopolymers

Figure 15 and Figure 16 show the results for the compressive and bending strengths for fly ash samples modified with CMC and PEG respectively. The base formula had an average compressive strength of 25 MPa with a bending strength of 3 MPa. When CMC was added these strengths remained almost constant at the 0.5% loading except at 1.0%. At the 1.0% loading the

compressive strength dropped to approximately 10% while the bending strength increased 30%. The formula with 1.5% CMC was not workable.

The addition of PEG had a negative effect in the compressive strength in range of loading studied but it showed a modest 17% improvement in the bending strength at 1.0%. At the 1.5% loading, both strengths dropped.

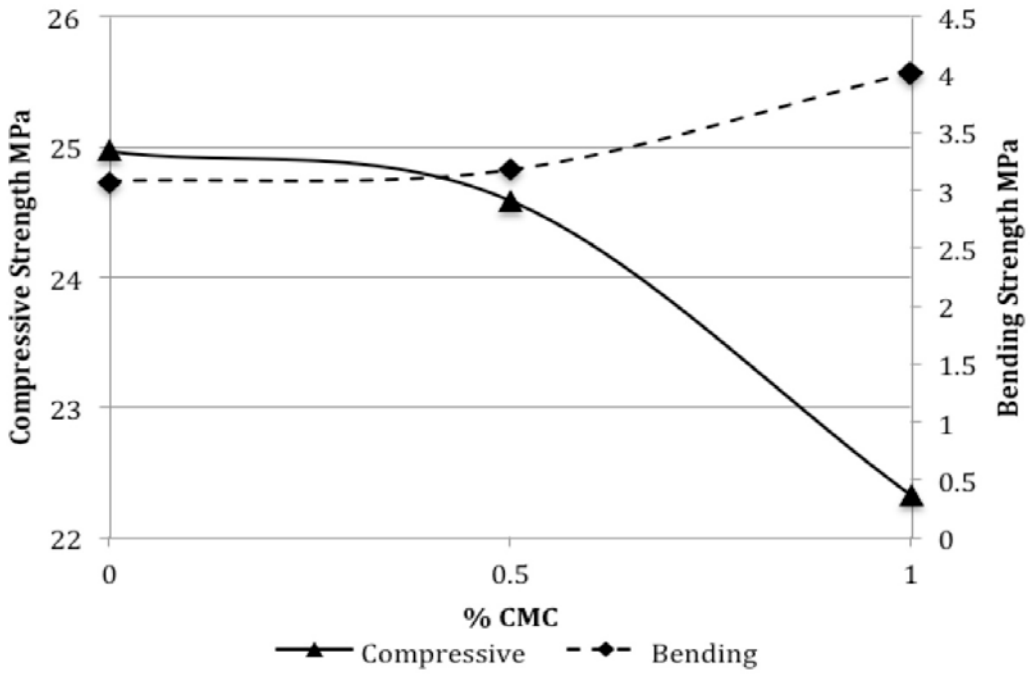

Figure 15. Average Compressive and Bending Strengths for CMC Fly Ash Geopolymer

All plates prepared for DMA analysis were very rigid and the instrument was not able to detect any significant changes in E', E" and Tan δ at any polymer loading. This may have been caused because fly ash plates were in average 2 times thicker than metakaolin plates under the same processing conditions, this could be the cause for which changes between CMC and PEG geopolymers were not detected

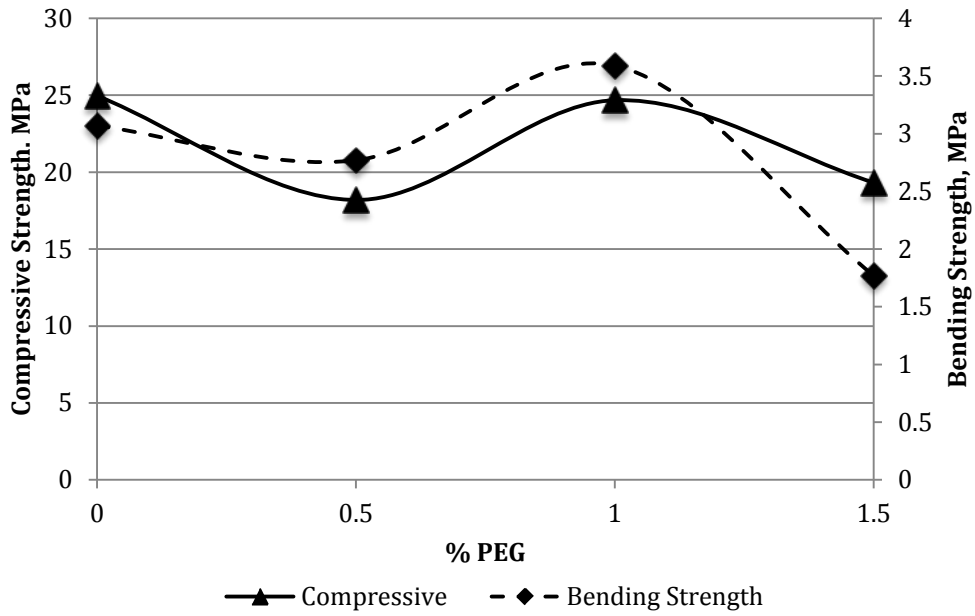

Figure 16. Average Compressive and Bending Strengths for PEG Fly Ash Geopolymers

Figure 17 presents a typical DMA result for fly ash samples. The behavior of elastic and viscous moduli with temperature is similar to the previously observed with metakaolin geopolymers, however, Tan δ rises sharply indicating that E' is decreasing faster than E". No transitions were detected in this temperature range.

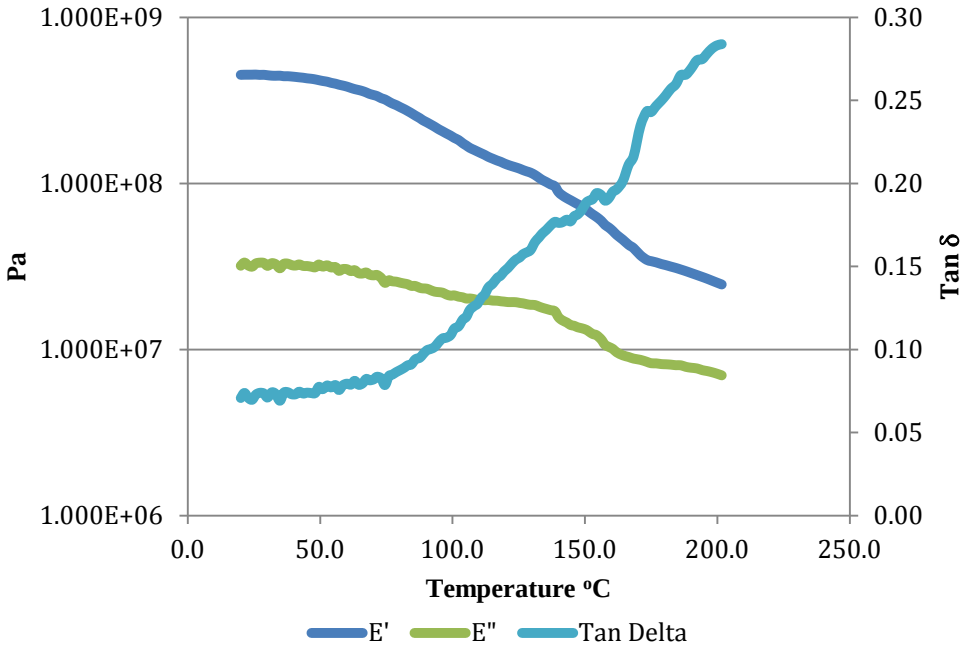

Figure 17. Typical DMA Plot Fly Ash Geopolymer with PEG or CMC

Figure 18 presents a SEM micrograph of the base fly ash geopolymer. The microstructure is composed of unreacted spherical particles of fly ash surrounded by an amorphous phase. When CMC was added, some needle like formations similar to metakaolin geopolymers were observed, as presented in Figure 19.

Figure 20 presents a micrograph of the fly ash geopolymer modified with PEG. For this formulation the microstructure is similar to the original fly ash formulation however it appears to be more compact.

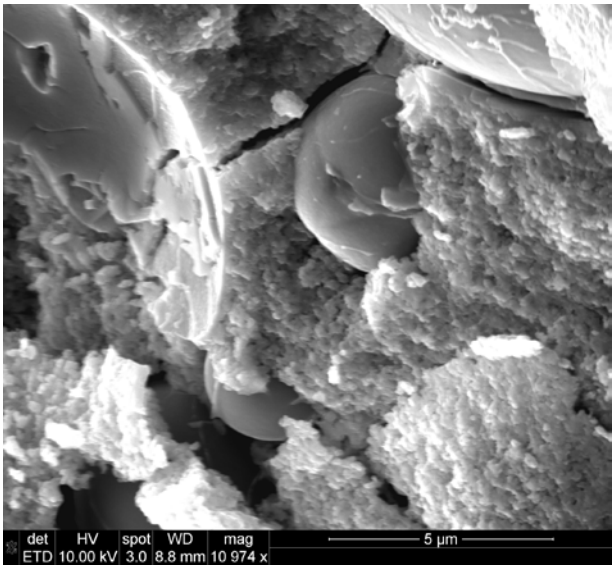

Figure 18. SEM Micrograph of Fly Ash Geopolymer

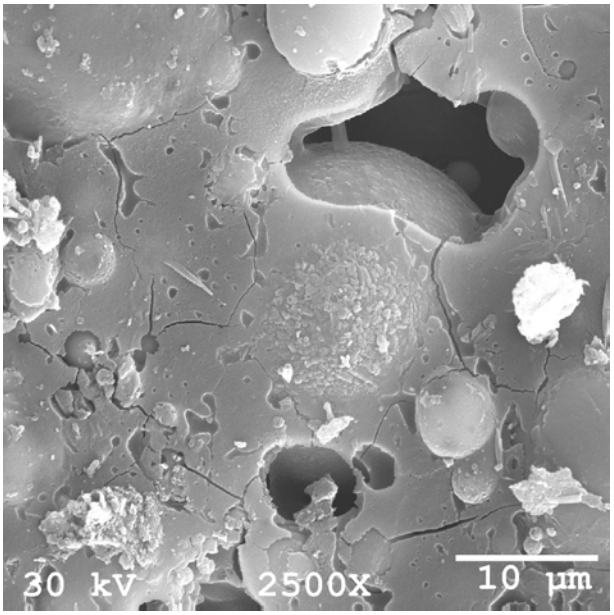

Figure 19. SEM Micrograph of Fly Ash Geopolymer with CMC

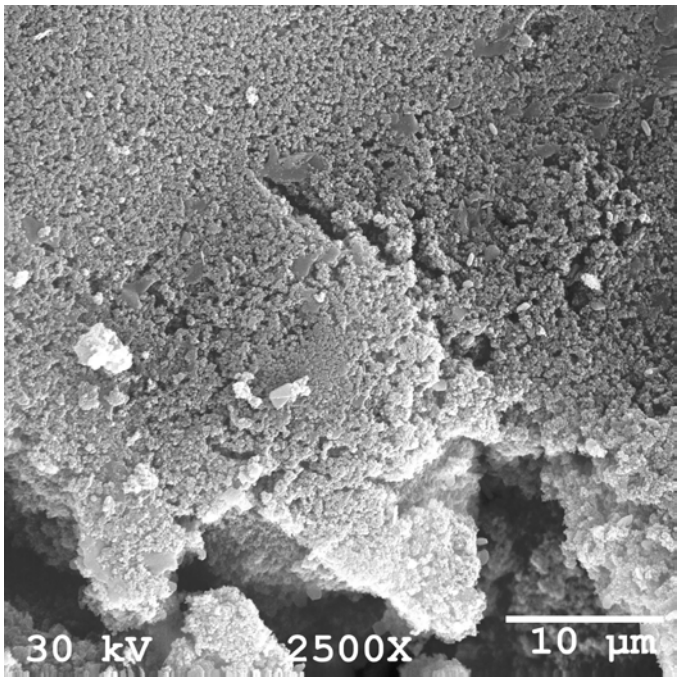

Figure 20. SEM micrograph of Fly Ash Geopolymer with PEG

4.3. Experiments with Higher Polymer Loading

In a second set of experiments we investigated the effect of higher loadings of CMC in the viscoelastic response of metakaolin geopolymer plates. An activating solution with the $SiO_2/Na_2O = 1.2$ and $H_2O/Na_2O = 15.5$ was prepared as previously described but this time the loading of CMC represented the percentage of the total mass of the solution. These plates were pressed as usual but cured at 85 °C. This temperature was chosen based on a recent report by T.M. Metroke [12] in which she explored the effect of curing conditions on the mechanical strength of geopolymers.

Figure 21, Figure 22 and Figure 23 present the measured values for E', E" and Tan δ respectively. Under these conditions, the plates shown an opposite effect with respect to the first set. At room temperature E' increased by a factor of 3 at a 0.5 % CMC loading while E" increased by a factor of 1.8. As the loading increased, both moduli attained similar values to the original formula. The Tan δ plot shows that this value decreases with CMC loading indicating that the change in E' dominates over E" however two transition at approximately 220 °C and 320 °C were observed.

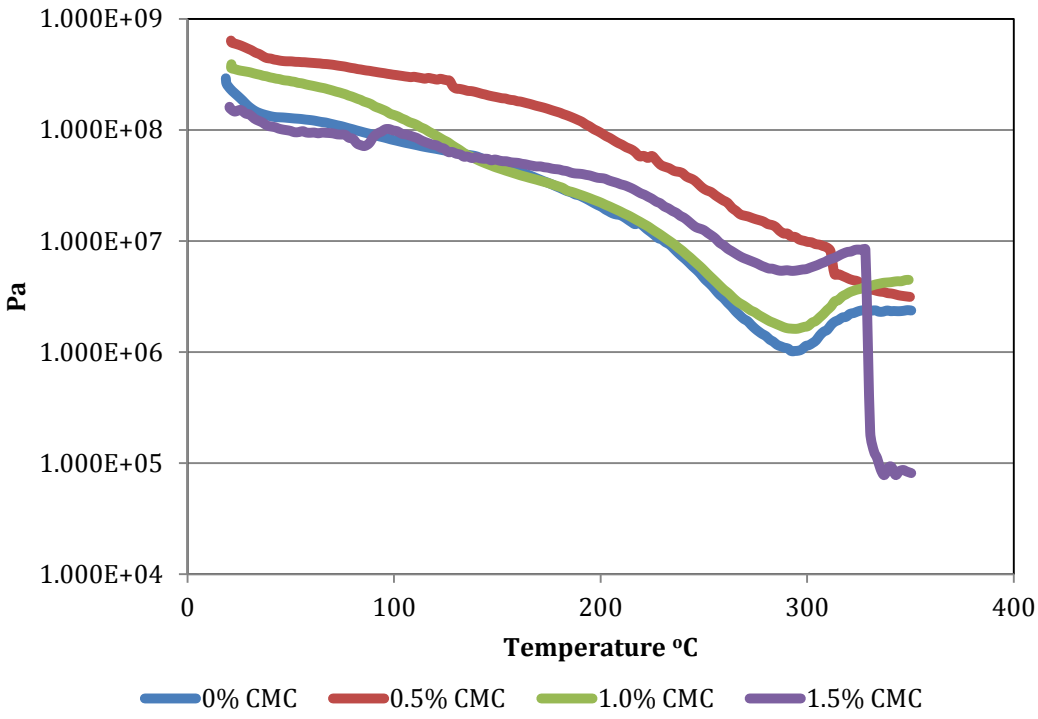

Figure 21. Elastic Modulus (E') for Metakaolin Geopolymer with CMC

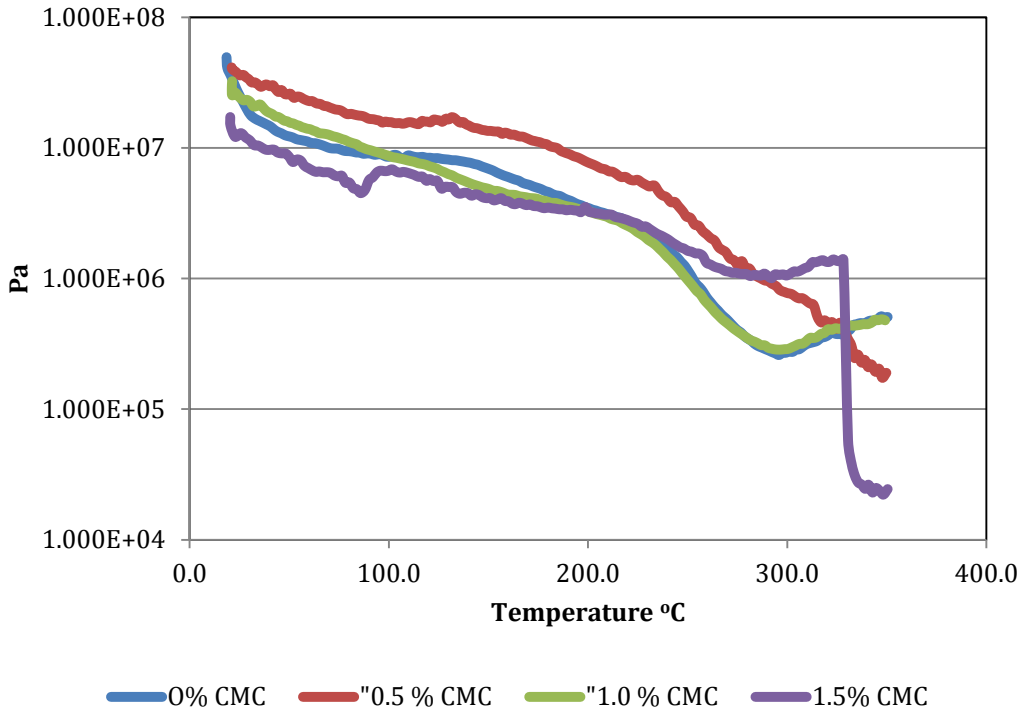

Figure 22. Viscous Modulus (E") for Metakaolin Geopolymer with CMC

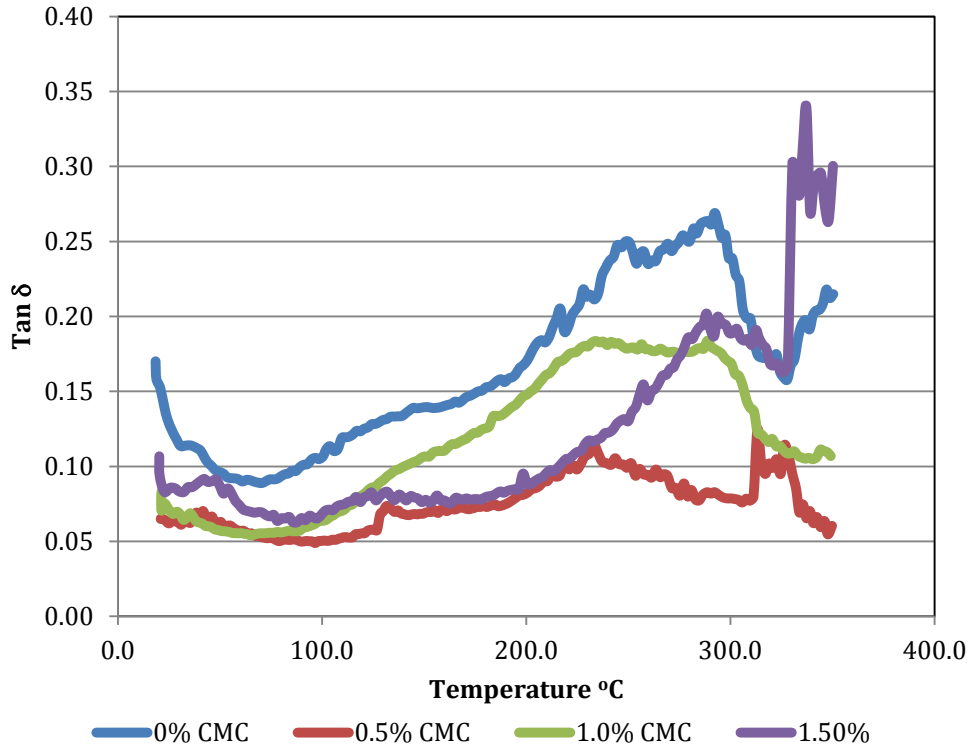

Figure 23. Tan δ for Metakaolin Geopolymer with CMC

Also in a second set of experiments we investigated the effect of adding more CMC and PEG to fly ash formulas. An activating solution with molar ratios of $SiO_2/Na_2O = 1$ and $H_2O/Na_2O = 13$ was prepared as described before. For these formulas the amount of dissolved polymer was calculated as mass percentage of the total activating solution. These samples were cured at 85 °C for 24 hours.

As seen in Figure 24, the compressive and bending strengths of the base formulation were 45.3 MPa and 3.8 MPa respectively. In average, the addition of CMC showed a similar trend as with the previous fly ash formulations. CMC causes the compressive strength to decrease but tends to increase the bending strength. At the 1.0 % CMC loading the average compressive strength was 34.7 MPa and he bending strength was 6.5 MPa.

A similar behavior was observed with PEG. As the amount of PEG increases the compressive strength decreases but the bending strength tends to increase. The best bending strength was 5.7 MPa and it was obtained with 1.0 % PEG. These results are presented in Figure 25.

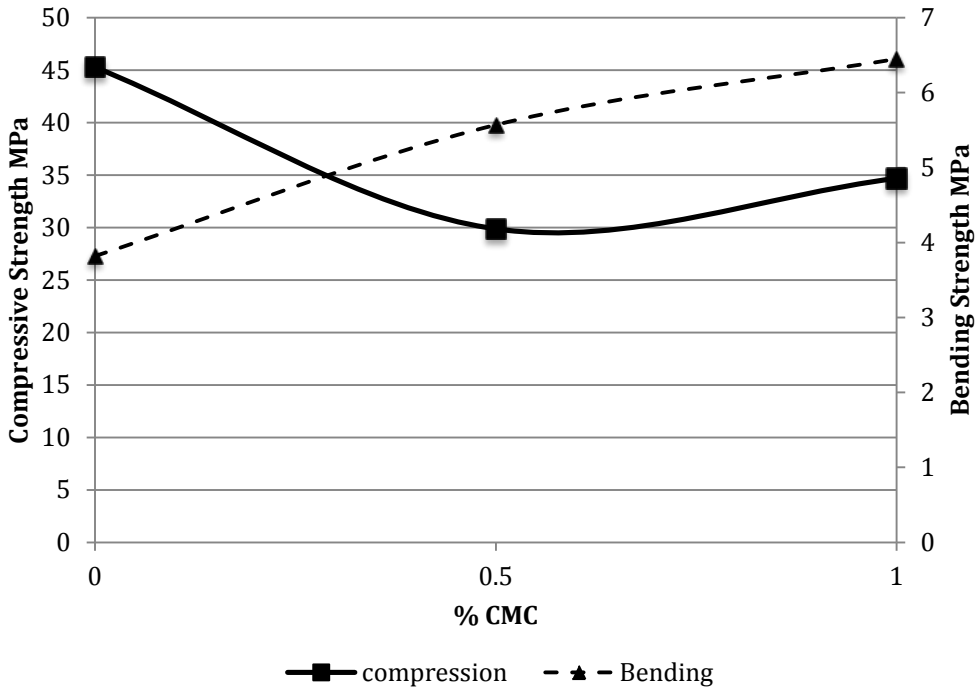

Figure 24. Effect of CMC in the Compressive and Bending Strengths of Fly Ash Geopolymer

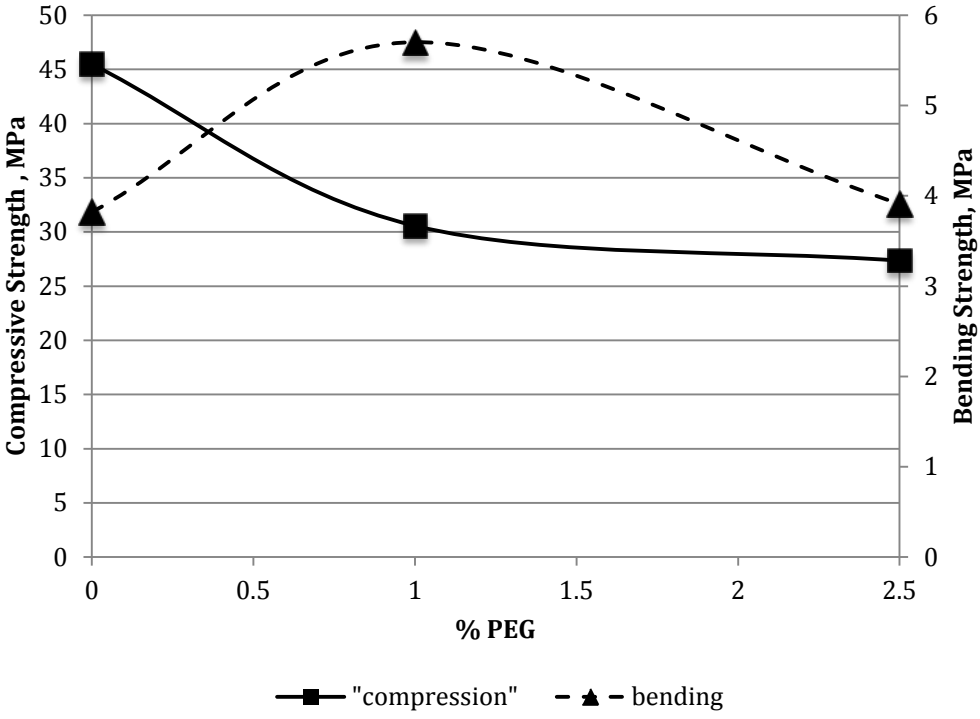

Figure 25. Effect of PEG in the Compressive and Bending Strength of Fly ash Geopolymers

4.4. Effect of Fibers

Another objective of this project was to conduct a preliminary evaluation of the effect of cellulose (cotton) and PVA fibers in the mechanical response of the polymer modified geopolymers. The idea was to see if two different randomly dispersed fibers would yield plates with similar flexibility to the ones with the cheesecloth mesh.

The cotton fibers were obtained by chopping the cheesecloth into strands of approximately 5 mm long. The PVA fibers used were RECS15 (Kuraray America) with a nominal length of 6 mm.

For both metakaolin and fly ash geopolymers the first formulation with 1 % CMC was chosen. This selection was based in the fact that flexibility was observed in metakaolin formulas with CMC and in both cases 1.0 % loading of CMC yielded the best bending strength.

The amount of fibers added represented the 1.5 volume percent of the total geopolymer mix. The fibers were first mixed with either the metakaolin or the fly ash in a rotary mill for 20 minutes to achieve a homogenous dispersion. The rest of the process was followed as described previously.

Table 2. Bending and Compressive Strength for Geopolymers with 1.5% Volume of Fibers

Geopolymer	Fiber	Compressive strength (MPa)	Bending Strength (MPa)
Metakaolin, 1% CMC	Cotton	40.5	3.4
Metakaolin, 1% CMC	PVA RECS15	42.8	9.4
Fly Ash 1% CMC	Cotton	39	5.9
Fly Ash 1% CMC	PVA RECS15	36.8	6.4*

* This value represents the average of 2 samples

For metakaolin geopolymers modified with CMC the use of cotton fibers increased the compressive by a factor of 1.5 while the bending strength remained at the same level. PVA fibers had a similar effect in the compressive strength but they changed the bending strength by a factor of 2.5.

In the case of fly ash geopolymers a similar effect to the cotton fibers was observed in the compressive strength, it increased by a factor of 1.7 while the bending strength increased by a factor of 1.5. When PVA fibers were added, both the compressive and bending strengths increased roughly by the same factor of 1.6.

In general the use of both fibers increased the compressive strength of both types of geopolymers by the same factor however PVA fibers had a dominant effect in the bending strength of

metakaolin geopolymers. It was also observed that the use of PVA fibers enhanced the ductility of both geopolymers.

Plates for DMA testing were manufactured by the same procedure as described above. These plates were either too rigid or too brittle that this test could not be performed.

5. CONCLUSIONS

From this preliminary series of experiments we can conclude that it is possible to manufacture geopolymer plates with enhanced flexibility. Geopolymers are brittle by nature but when a solution of CMC was added to the metakaolin geopolymer mix, the plates obtained showed enhanced flexibility. Results indicate that a combination of extra water and CMC is required to achieve these results. While it was observed that adding extra water to the plates will add flexibility, it was also observed that these samples tend to shrink and crack with time. This effect was not observed in the plates made with an addition of CMC solution. To achieve flexibility, the loading of CMC in the solution should be in the range of 0.5% – 1.0% per mass.

DMA indicates that the addition of a 0.5% CMC solution decreased the Elastic Modulus by 75%, which causes the samples to be less rigid. As the percentage of CMC was increased, the damping of the geopolymer (Tan δ) increased 75%, which could result in the enhancement of the impact properties of the geopolymer. The addition of 1.0% PEG modified significantly the compressive strength of metakaolin geopolymers but not its bending strength.

Fly ash formulations did not show the same flexibility as observed with metakaolin. A possible cause is that fly ash particles are much coarser. Under the same processing conditions the plates with fly ash were much thicker than the metakaolin ones. In both sets of experiments it was observed that the addition of CMC enhances the bending strength of fly ash geopolymers while in average the compressive strength tends to decrease.

The addition of fibers to geopolymers modified both compressive and bending strength and added flexibility to the tested beams, however this flexibility was not observed in the plates.

6. RECOMMENDATIONS

The addition of CMC and PEG solutions to geopolymers has demonstrated that the mechanical response of metakaolin and fly ash geopolymers can be modified. The results presented in this report show a trend based on a limited number of samples. It is recommended to prepare and test more samples to corroborate these observations and evaluate the deflection at the center of the beams during three point bending test.

The change in Tan δ with the addition of CMC solution to metakaolin geopolymers is a phenomenon that could be studied further more by impact testing. It is also recommended to prepare more samples of metakaolin geopolymer with higher content of CMC Solution (more than 22.5%) and evaluate their flexibility and durability under different service conditions, such as high temperature and thermal cycling.

For the case of fly ash geopolymers, DMA analysis didn't show changes in E' and E" because the samples were too thick for the instrument but it is possible to measure these parameters with ultrasound. The increase of the bending strength with higher loading of CMC in fly ash geopolymers is an effect worth of future studies.

The last two recommendations are to evaluate the effect of these polymers in the setting and rheology properties and evaluate their mechanical response when exposed to high temperatures.

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LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS

AFRL	Air Force Research Laboratory, Airbase Technology Division
CMC	carboxymethyl cellulose
DMA	dynamic mechanical analysis
E’	elastic modulus
E”	viscous modulus
MW	molecular weight
PEG	polyethylene glycol
PI	project investigator
PVA	polyvinyl alcohol
SEM	scanning electron microscope
Tan δ	damping